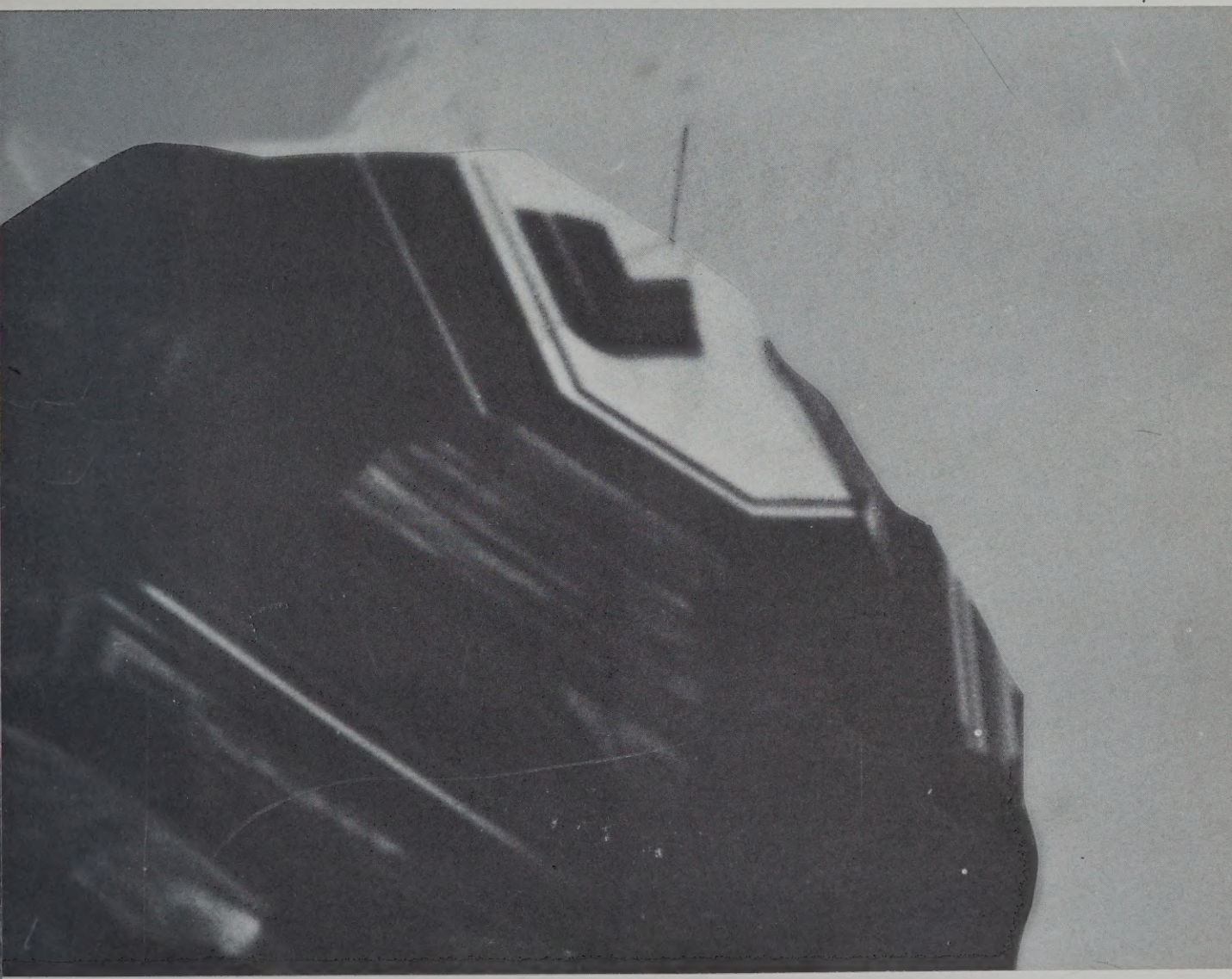
NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN



VOLUME 45 AUGUST 1961 NUMBER 8

U.S. DEPARTMENT OF COMMERCE — LUTHER H. HODGES, *Secretary*

NATIONAL BUREAU OF STANDARDS — A. V. ASTIN, *Director*

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Technical News

BULLETIN

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COVER: A zinc crystal grown from vapor in recent experiments at the National Bureau of Standards. Stepped prism planes have formed around the sides of this crystal, and a tiny whisker has grown upward from the basal (upper) plane. (× 69.) Investigation of crystal growth processes is fundamental to studies of the properties of metallurgical materials. See "Growth of Metal Crystals From the Vapor Phase," p. 130.

High-Flux Research Reactor
for New NBS Site

DESIGN WORK is now nearly complete for a high-flux research reactor to be constructed on the Bureau's new site at Gaithersburg, Md. The reactor, to be known as the NBSR, and its associated laboratories [1] will provide a variety of neutron and gamma-ray sources for use by the Bureau and other Government agencies in the Washington area having the same general requirements.

The new reactor will enable the Bureau to fulfill its growing responsibilities in the many rapidly expanding fields of atomic energy. For example, the Bureau has developed a low-intensity neutron standard that makes it possible for all laboratories to measure neutron radiation intensities in terms of a common reference value; however, it has lacked facilities for measuring high-intensity fluxes such as occur in a nuclear reactor. Reliable precision measurement techniques and standards for high- and low-energy neutron fluxes are also urgently needed both in the power reactor field and in various areas of scientific or applied research, such as radiation effects and health physics instrumentation.

Perhaps the most significant function of the NBSR will be its use in studying certain fundamental properties of matter. The intense thermal beams provided by the reactor will constitute a powerful tool in the analysis of atomic and molecular structure by neutron diffraction. This recent technique can be applied to investigate various aspects of crystal structure, such as the location of hydrogen atoms, magnetic crystal properties, intermolecular force constants, and chemical bond strength. Other structures of interest are those of liquids and vitreous materials.

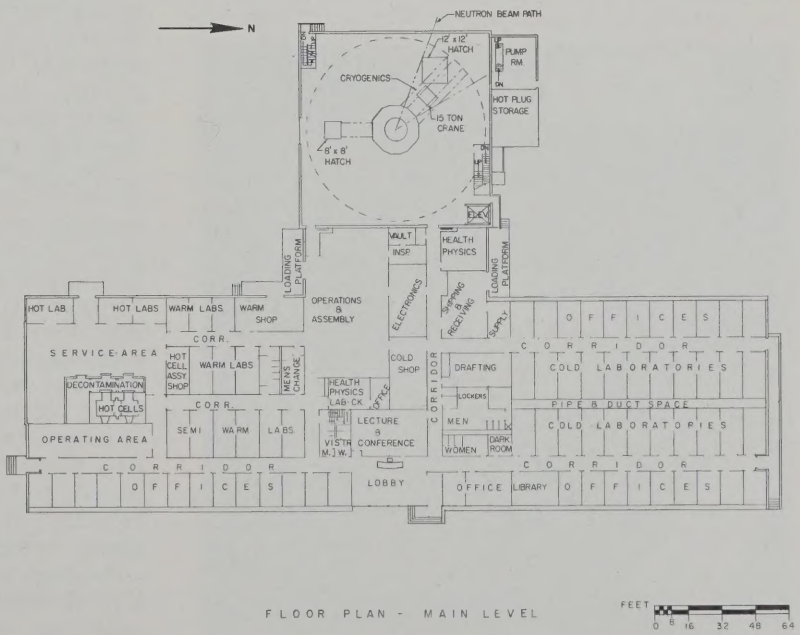
The reactor will advance the measurement, analysis, and understanding of the effects of radiation on substances of all kinds. Such information is of great value in solid state and chemical physics, and in such practical engineering problems as radiation processing and the effects of radiation on structural materials.

Besides providing radioisotopes for standards of radioactivity, the reactor will supply radioactive tracers for the investigation of such phenomena as diffusion (e.g., self-diffusion in metals), exchange (e.g., chemical exchange), phase transformation (e.g., sublimation), and complex flow (e.g., turbulent flow and multiphase flow). Neutrons will be provided for chemical analysis by the measurement of nuclear decay products resulting from neutron absorption. This latter technique is of extremely wide applicability and extends even to the analysis of anthropological artifacts.

Of particular importance among the basic processes to be studied is that of fission. Inadequate understanding of this process and lack of information on neutron yields still limits the design of fissile material breeding plants.

The NBSR will be similar to the CP5 research reactor at the Argonne National Laboratory near Chicago, Ill., in that it will be a tank-type reactor using enriched fuel and heavy water moderator. The initial operating level will approach 10 Mw; fixed features will be designed to permit operation at 20 to 25 Mw when additional cooling towers and heat exchangers are added. The initial operating power will provide an in-pile flux of 10^{14} neutrons per square centimeter per second.

The 24 fuel assemblies of the NBSR will be of uranium-aluminum alloy clad with aluminum. Each assembly will be composed of 17 fuel plates and, although continuous, will have two active portions separated by a dummy aluminum inactive section. This construction permits continuous coolant flow along the entire



Plan of the building that will contain the Bureau's high-flux research reactor to be constructed at Gaithersburg, Md.

FLOOR PLAN - MAIN LEVEL

length of the assembly, but eliminates fuel in the central plane of the pile. It has the effect of reducing the fast neutron and gamma-ray components emanating from the horizontal beam ports.

Specially constructed converter fuel elements will provide fast neutron in-core irradiation space. Instrumented samples will be placed in this space for basic studies of fast neutron effects. In at least one of these regions cryogenic equipment will be employed to maintain near-liquid-helium temperatures during sample exposure.

Plans are progressing for an in-pile cold neutron moderator that will provide the low-energy neutron flux required to study the problem of crystal lattice vibrations and a variety of long-range order problems. As the neutron slowing-down process from thermal to subthermal energies is inadequately understood at present, the Bureau plans to investigate this problem experimentally before the final design of this particular apparatus is completed.

Associated with the reactor will be a complete complex containing shops, offices, cold laboratories (nonradioactive areas for assembly and servicing of equipment to be used at reactor beam ports or in conjunction with in-pile experimentation), warm laboratories (moderately radioactive areas for examination of intermediate level radioactive samples or chemical processes), and hot cells (highly radioactive areas for examination of very radioactive samples or chemical reactions).

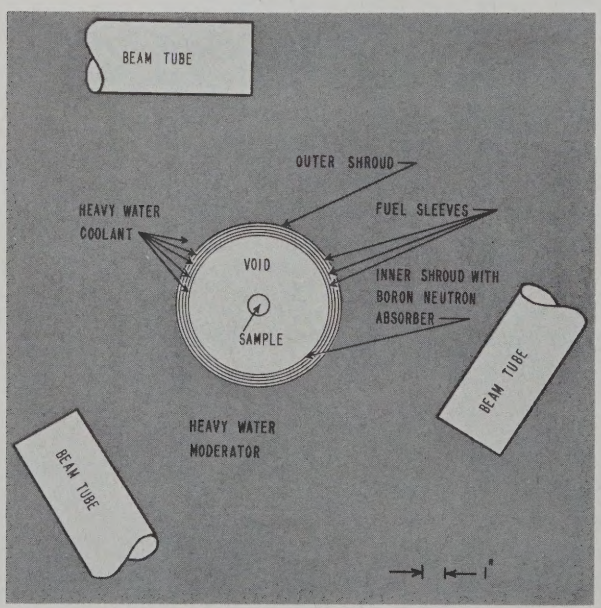
[1] *Design architects are Burns & Roe, Inc., of New York City.*

The Black Void Reactor Concept

THE BUREAU has been investigating a promising concept in research reactor design—that of the Black Void Reactor,[1] or BVR. The BVR can simultaneously provide both fast and slow neutron fluxes that are spatially separated. An arrangement that provides this separation, conceived by C. O. Muehlhause, would permit radiation effects studies and thermal neutron beam studies (e.g., neutron diffraction) to be conducted independently in the same reactor. In addition, this arrangement constitutes a type of flux trap (inverted) which yields higher fluxes for a given power input than does the usual research reactor.

During a design study for the National Bureau of Standards Reactor (NBSR),[2] to be constructed at the new NBS site at Gaithersburg, Md., consideration was given to the idea of incorporating a converter assembly into the NBSR design. This consideration led to the BVR concept of reactor design. Although the BVR appears to possess a number of advantages, it requires significant fuel element development and testing before it can be applied. Two new reactor types[3] under consideration by the Atomic Energy Commission may contribute technique to the development of the heavily loaded annular fuel elements required for the BVR.

The 8-in.-diameter BVR–1. The sample to be irradiated by fission neutrons is placed in the void space. A limited number of beam tubes may be inserted in the moderator. The probability of neutron return is 0.543.



The general type of Black Void Reactor is the BVR–N, where N represents the number of heavily loaded (appearing black to thermal neutrons) cylindrical fuel assemblies in the configuration. As in other reactors, the fast neutrons released by fission in the fuel walls are slowed by the moderator, and a certain portion of these now slow, or thermal, neutrons return to the fuel assembly. Some of these thermal neutrons will be absorbed by the fuel walls and converted into fast fission neutrons to maintain the chain reaction.

In the BVR, a portion of the fast fission neutrons and some neutrons of intermediate energy that have been partially moderated irradiate the void interior of the tube. A sizable space is thus provided in which instrumented samples may be placed for radiation effects studies. At the same time, beam tubes in the moderator outside the fuel assemblies can provide thermal neutrons for use in neutron diffraction studies.

The simplest approach to the Black Void Reactor is the BVR–1, consisting of a single long cylindrical fuel assembly immersed in a large tank of moderating material. A practical example would be an assembly 8 ft in length and 8 in. in diameter. According to the design study, fast neutrons leaving the fuel walls would slow down in the moderator and return to the walls with a probability of 0.543. The overall multiplication of the system (number of fast fission neutrons produced per neutron absorbed in the fuel walls) must be adjusted to have the inverse of this probability value. At a power of 3 Mw, a fast neutron flux of approximately $10^{14}/cm^2$ sec would be available within the cylinder. A limited number of beam tubes may be placed in the moderator of the BVR–1.

Another example of the BVR is the symmetrical BVR–3, which comprises three 6-in.-diameter, 8-ft-long cylindrical assemblies spaced 1.73 ft apart. Here the

The symmetrical 6-in.-diameter BVR–3. Three sample spaces are available for fast neutron irradiation. The probability of neutron return is 0.642.

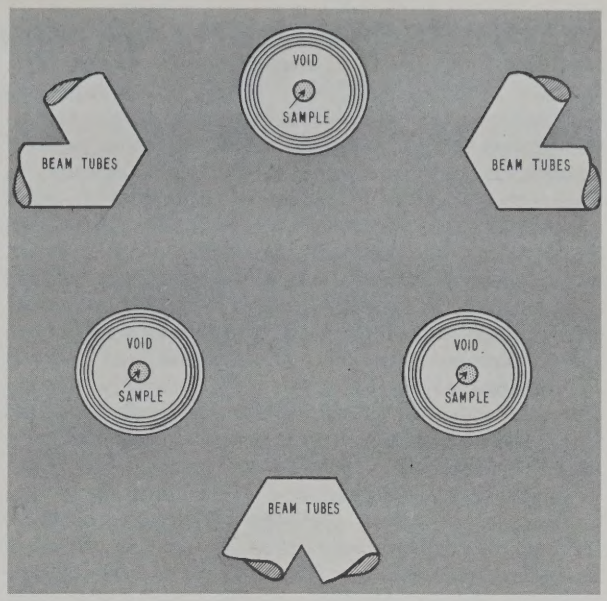
return probability for neutrons leaving one assembly and being absorbed in any of the three assemblies is 0.642. Thus, the walls of each assembly must have a multiplication in excess of 1.56.

The design study showed that as a research reactor, the BVR should have the advantages of long assembly life (greater than 1 yr at several megawatts) and safe operation due to long mean neutron lifetime. There is adequate excess reactivity for the thermal neutron beam tubes. The beams may be readily aimed at the space between assemblies and thus avoid virgin fission neutrons and gamma rays.

[1] *For further information, see* The black void reactor concept, *by C. O. Muehlhause, Nuclear Sci. and Eng.* **7**, *505 (June 1960).*

[2] *See* High-flux research reactor for new NBS site, *p. 127, this issue.*

[3] *High Flux Beam Reactor at Brookhaven National Laboratory, and High Flux Isotope Reactor at Oak Ridge National Laboratory.*



Polymer Crystals With Folded Chains

THE NATURE of polymer crystals is of considerable interest, since such crystals affect the mechanical, optical, and dielectric properties of these useful materials. As part of a long-range program involving the dielectric properties of polymers, scientists at the Bureau have investigated the fundamental characteristics [1] of these materials from a theoretical point of view.

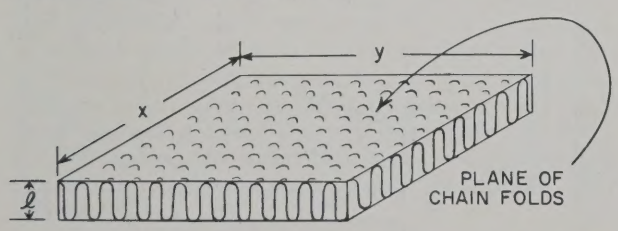
In 1958, it was discovered that certain polymers, such as polyethylene, are deposited from dilute solution in the form of flat and very thin crystalline platelets. (See, for example, the work of A. Keller and coworkers.[2, 3]) The surprising feature of these platelike crystals is that they are built up of *folded* polymer chains. The folds themselves actually form the large upper and lower surfaces of the platelets. The chain axes of the unfolded portions of the polymer molecules are roughly perpendicular to the plane of the chain folds. This is a vastly different structure from the bundlelike or sheaflike structure that one might have expected the crystals to exhibit.

A detailed theoretical interpretation of this phenomenon has recently been presented by J. I. Lauritzen and J. D. Hoffman of the dielectrics laboratory. Their explanation involves nucleation and growth theory. They have demonstrated that in sufficiently dilute solution, the rate of formation of the seeds leading to folded crystals must exceed that of seeds leading to crystals of the bundlelike type.

The growth of each folded nucleus was then considered. It was shown that the thickness (often called the step height, l^*) will not increase rapidly as the platelet becomes larger in the x- and y-directions, thus preserving the thin platelet character of the crystal. This tendency for the step height to maintain itself during the growth process is a result of the fact that a crystal of step height l^* grows much faster than one that is either substantially thicker or thinner. The quantity l^* generally ranges from roughly 100 to 500 A, and increases with temperature of crystallization.

The theoretical study has recently been extended to the case of crystallization in bulk polymers.[4] Results were obtained which strongly suggest that substantially chain-folded crystals (lamellae) exist in bulk polymers, especially in the spherulites. Calculations have recently been made at the Bureau which give information on the smoothness of the chain-folded surfaces of the lamellae and on the details of the melting behavior to be expected for chain-folded crystals.[5]

Details of loop-type polyethylene crystal formed from dilute solution. Step height is l^*.



[1] *For further information, see* Theory of formation of polymer crystals with folded chains in dilute solution, *by John I. Lauritzen, Jr., and John D. Hoffman, NBS J. Research* **64A** *(Phys. and Chem.), No. 1, 73 (1960).*

[2] *A. Keller and A. O'Connor,* Discussions, *Faraday Soc.* **25**, *114 (1958).*

[3] *A. Keller, Phil. Mag.* **2**, *1171 (1957).*

[4] Theory of rate of lamellar spherulitic crystallization in bulk polymers, *by John D. Hoffman and J. I. Lauritzen, Jr., NBS J. Research* **65A** *(Phys. and Chem.), No. 4, 297 (1961).*

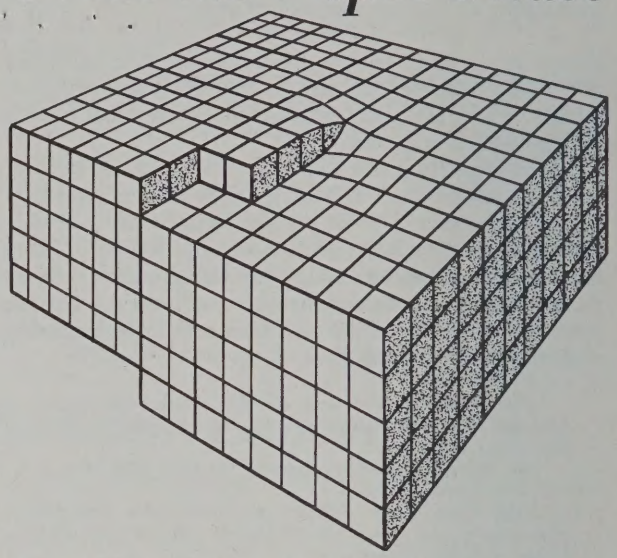
[5] The melting process and the equilibrium melting temperature of polychlorotrifluoroethylene, *by J. D. Hoffman and J. J. Weeks (to be published 1961).*

Growth of Metal Crystals From the Vapor Phase

IN A BUREAU PROGRAM of fundamental research in metal physics, a study was recently made of the growth of zinc crystals from the vapor phase.[1] Growth rate data from experiments conducted by R. L. Parker and L. M. Kushner were found to be in good qualitative agreement with the screw dislocation theory [2] of crystal growth as modified by impurities, and to show no evidence of surface nucleation effects.[3] These results, among the first of this type to be obtained on monatomic systems, provide information on the mechanisms involved in the metallurgically important process of crystallization. They will be of considerable value in other studies now under way on the growth kinetics of potassium crystals and of mercury "whiskers."

In the transformation of a metal from the vapor phase to a crystalline solid, the velocity of growth of the crystal surface depends on the driving force of vapor supersaturation. One theory [2] of this velocity-supersaturation dependence assumes that a screw dislocation emerges on the crystal surface to provide a monatomic step for continuous crystal growth at low supersaturations. In another theory,[3] new layers are started by the formation of two-dimensional nuclei on the surface of a completed layer as a result of spontaneous fluctuations in the surface density of adsorbed atoms, and relatively high vapor supersaturations are required to achieve this nucleation. In the present work to provide experimental data for the evaluation of these two theories, the growth rate of zinc crystals was investigated at low supersaturations (near equilibrium), since the rate predictions of the two concepts differ primarily in this area.

Zinc was chosen for the study because of its monatomic vapor, its high vapor pressure at convenient temperatures, and its availability in quite high purity (99.999+ percent). In the experiments, the crystals were nucleated and grown in a Pyrex tube, about 13 cm long and 2.5 cm in diameter. A tungsten rod, sealed into one end of this "growth" tube, served as a cold tip on which nucleation and crystal formation could take place.

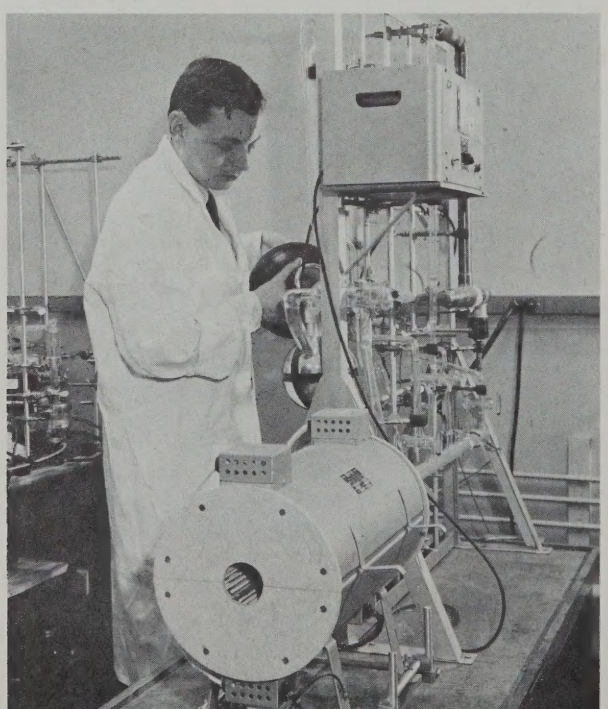


Screw dislocation model which provides a monatomic step for continuous crystal growth. Recent NBS experiments support the theory of crystallization illustrated by this model.

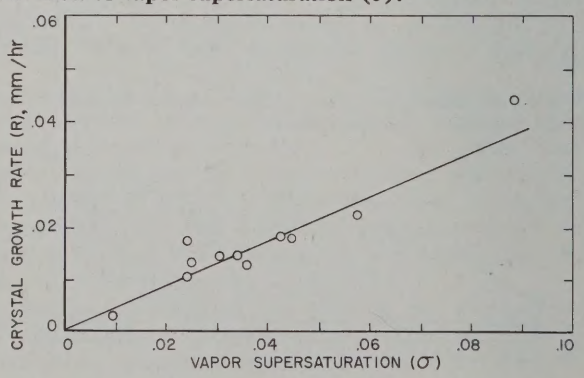
In preparation for the experiments, the growth tube was sealed, together with a Pyrex connecting tube containing approximately 10 g of zinc, to a high vacuum system that previously had been outgassed for 18 hr at 400 °C. A portion of the connecting tube was then fitted inside an electric tubular furnace and heated to 400 °C. When several grams of the zinc had distilled from this tube into the cold growth tube, the latter was sealed off from the vacuum system.

Growth rate measurements were obtained by inserting the growth tube into an apparatus which provided for continuous weighing of the crystal as it grew in supersaturated vapor. In this apparatus, consisting essentially of a large oven in which the proper tempera-

R. L. Parker fills a liquid nitrogen trap in a high-vacuum system used to prepare the tubes in which metal crystals are grown from the vapor phase. In the foreground is an electric tubular furnace for distilling the zinc vapor.



The rate of crystal growth (R) from vapor is shown as a function of vapor supersaturation (σ).



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ture and supersaturation could be accurately maintained, the tube was mounted on a pivot above its center of gravity in such a manner that the end of the tube containing the tungsten rod tilted downward as the crystal grew. An automatic weighing device, activated by a photocell, was used to plot weight vs time on a recorder.

During the experiments, the growth tube was kept at a temperature T and the tungsten rod at a temperature $T_0 < T$ by means of a controlled heat leak fitted into the oven. The crystal was nucleated on the tungsten rod by gradually increasing the temperature difference ΔT. After nucleation occurred, ΔT was adjusted to a value which determined the vapor supersaturation σ, and the weight-vs-time measurements were recorded. This procedure was repeated at various ΔT values, and the weight-time information thus obtained was reduced to growth-velocity data.

The experiments showed that zinc crystals, in the form of truncated hexagonal pyramids, grow at supersaturations much less than the critical value established by the surface nucleation theory. Below this value, $\sigma_c \approx 0.8$, the theory predicts that growth virtually ceases. Furthermore, the nucleation upon the tungsten rod occurs at $\sigma \approx 0.02$ to 0.03, which is also much lower than expected from nucleation theory. However, when the experimental data were compared with the rate parameters of the screw dislocation theory, good agreement was found.

The effects of impurities on the growth rate were calculated from estimates of the rate of diffusion of water molecules (probably the major source of impurity) from the bulk of the Pyrex glass, and of their adsorption to the zinc crystal surface. According to a recent theory,[4] a partial reduction in step velocity may be expected for a given surface concentration of immobile surface impurities. The Bureau calculations showed some evidence of this effect.

Results thus far obtained in experiments with very pure potassium crystals distilled in an ultra-high vacuum are in contrast to those of other investigators,[5] who found that surface nucleation appears to limit the crystallization velocity of potassium. The Bureau study, although not yet complete, does not show such condensation lags, even at low supersaturations.

The mercury whiskers now being investigated are single crystals of only 100 A or so in thickness, and from 5 to 50 μ in length. Because these whiskers are extremely strong single crystals, they may be observed by an electron field-emission microscope during growth. At a magnification of about 300,000 ×, their growth velocity is being measured directly with this technique. To obtain quantitative information on the effect of impurities on crystal growth, future work will be undertaken to measure the growth kinetics of metal crystals in the presence of known amounts of foreign gas molecules.

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[1] *For further technical details, see* Growth rates of zinc crystals from the vapor phase, *by R. L. Parker and L. M. Kushner, J. Chem. Phys.* **35,** *No. 4 (Oct. 1961).*

[2] The growth of crystals and the equilibrium structure of their surfaces, *by W. K. Burton, N. Cabrera, and F. C. Frank, Trans. Roy. Soc. (London)* **243,** *299 (1951).*

[3] Kinetische Behandlung der Keimbildung in übersättigten Dämpfen, *by R. Becker and W. Döring, Ann. Phys.* **24,** *719 (1935).*

[4] *See N. Cabrera and D. Vermilyea in* "Growth and Perfection of Crystals," *edited by R. H. Doremus, B. W. Roberts, and D. Turnbull, Wiley, New York (1958), p. 393.*

[5] Die Verdampfungs- und Kondensationgeschwindigkeit des festen Kaliums, *by F. Hock and K. Neumann, Z. phys. Chem.* **2,** *241 (1954).*

Commerce Secretary Breaks Ground

for New NBS Laboratories

On June 14 at 10:30 a.m., Secretary of Commerce Luther H. Hodges broke ground for the new National Bureau of Standards laboratories at Gaithersburg, Md. The ceremony signaled the initial construction phase for a 20-building, $104 million research facility which will permit complete relocation of the Bureau from Northwest Washington to the 555-acre site in Montgomery County. On the platform behind the Secretary are NBS Director A. V. Astin, left, and John L. Moore, Administrator, General Services Administration.

"At no time in our history have the measurement needs of the country been greater," Secretary Hodges said at the ground-breaking ceremony. "The exploration of outer space, the growth of nuclear technology, the requirements of national defense, the rapid application of scientific developments to civilian technology, all of these factors point to a future in which the science of measurement will play an increasingly vital role. With the most modern facilities, the scientists and engineers of the National Bureau of Standards will wage the fight to keep the Nation's measurement competence abreast of the pressing demands of science and technology."

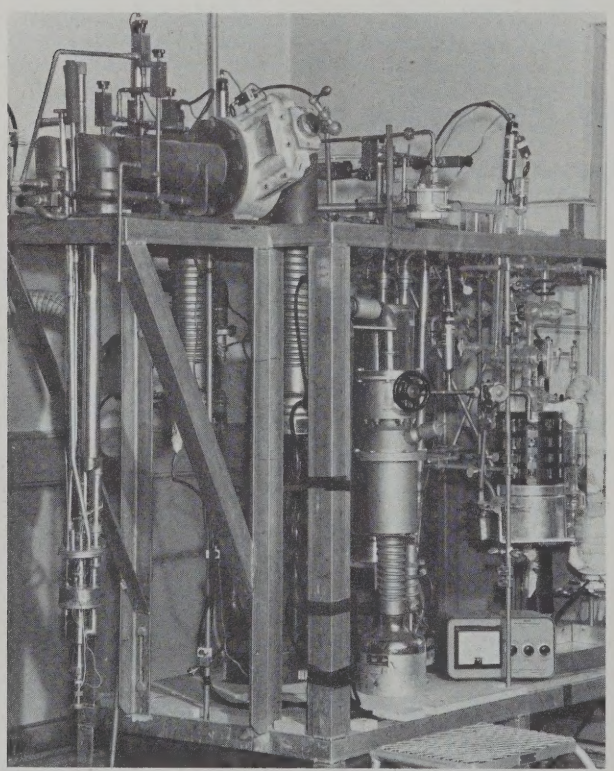
The gold-plated shovel the Secretary is using was originally used in 1915 to break ground for the Bureau's present Chemistry Building.

PORTABLE He^3 REFRIGERATOR

A CONTINUOUSLY OPERATING He^3 refrigerator, capable of maintaining temperatures as low as 0.26 °K, has been developed at the Bureau. This apparatus, designed by E. Ambler and R. Dove,[1] will be used for a variety of experiments, including magnetic and thermal measurements on paramagnetic substances, and nuclear orientation studies. The apparatus extends the region of convenient and continuous refrigeration down from about 1 °K, reached simply by using He^4, into the range previously accessible only by using the process of magnetic cooling. Although the refrigerator is not the first of its general type, it is particularly efficient and effective, and possesses many novel features which permit simple construction and operation.

The advantages of He^3 (isotopic abundance about 10^{-9}) over the abundant isotope He^4 for producing low temperatures have been recognized for some time. Lower temperatures can be produced by evaporating He^3 under reduced pressure for two reasons: The vapor pressure is higher at any given temperature, and He^3 exhibits no superfluid "film flow." In the case of He^4 the superfluid film creeps up the walls of the pumping tube and evaporates in the warmer regions. The resulting evolved gas must be pumped away, and its evaporation contributes no useful refrigeration.

He^3 refrigerator developed for maintaining temperatures as low as 0.26 °K. The exposed evaporating unit is seen at the lower left. A mercury ejector pump (center), without mechanical backing pump, is used to pump the He^3. This refrigerator is a prototype of one to be used in experiments with oriented nuclei.



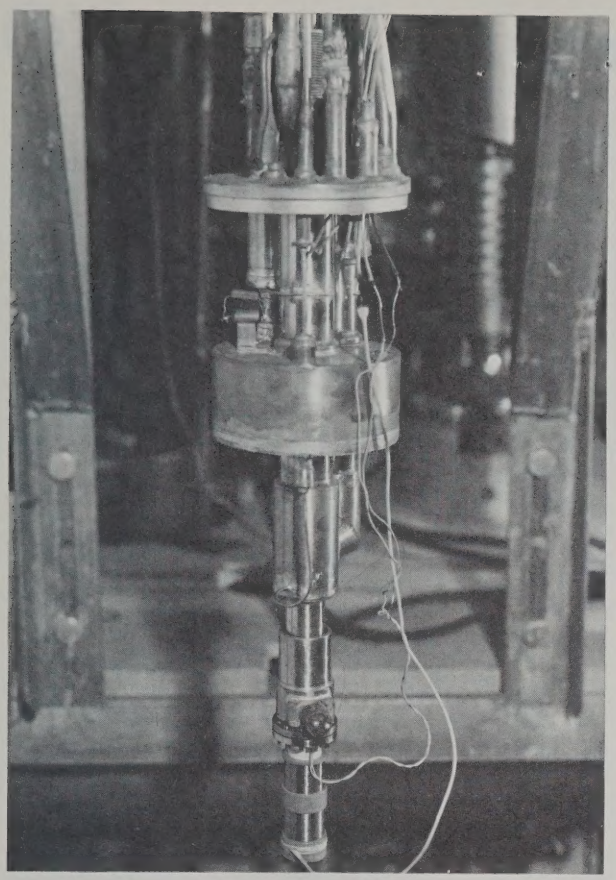
The rarity and high cost of He^3 requires that it be conserved, rather than be boiled off into the atmosphere as is He^4. As the present refrigerator uses a circulation system similar in some ways to that used in a conventional household refrigerator, only a small amount of He^3 is needed, and at the same time refrigeration can be obtained for as long as is desired.

To circulate the He^3, the refrigerator uses a mercury ejector pump (without a mechanical backing pump) operating at a discharge pressure of about 35 mm Hg. He^3 gas returning from the pumps is reliquefied in a condenser maintained at 0.8 °K by means of a pumped He^4 bath.

A porous stainless-steel plug acts as an automatic throttle for the condensed liquid returning to the evaporator. The pressure in the condenser, and hence across the plug, varies with the circulation rate so that there is automatic adjustment for different heat inputs. That is, as the heat input increases the evaporation rate of He^3 increases, and the pressure in the condenser rises as gas returns at a greater rate. This increase in pressure appears across the throttle, which can then accommodate a larger flow of liquid. With this arrangement there are no troublesome vapor locks.

The condenser, evaporator, and effective working space of the refrigerator are enclosed in a vacuum case and surrounded by a Dewar of He^4, which in turn is surrounded by a Dewar of nitrogen. Two cubic centimeters of liquid He^3 are used in this system. This volume proved to be more than sufficient, although it is felt that with suitable modifications of the present apparatus a similar temperature could be maintained with an even smaller volume of He^3. The He^3 is cleaned on each cycle by passing through liquid nitrogen cooled traps on either side of the mercury pump, and by a small trap, filled with copper wire, in the He^4 Dewar. The fact that no mechanical pumps are used allows a compact, fast, quiet, self-regulating system to be constructed.

The lowest temperature reached with this refrigerator is 0.26 °K. at zero heat input. The maximum refrigeration capacity is 2 mw, at which load the temperature can be maintained at 0.40 °K. These temperatures are achieved in a working volume of about 500 cm^3, and are measured by two independent methods. Vapor pressure measurements, taken with a McLeod gage and corrected for thermomolecular pressure, provide one temperature check, and a "magnetic thermom-



Evaporator used in the He^3 refrigerator. Gaseous helium returning from the pumps is liquefied in a condenser cooled by a pumped He^4 bath (drum in center). The liquid He^3 then passes downward through the thin tube into the evaporator, where it evaporates through a porous stainless steel plug into a space evacuated by a mercury ejector pump. The coils at the bottom are part of the apparatus used to determine the operating temperature of the refrigerator. This is done by determining the change in magnetic susceptibility of an enclosed sphere of chromic methylammonium alum. When the refrigerator is operating, this exposed portion would be surrounded first by a vacuum jacket, then by a He^4 Dewar, and finally by one containing liquid nitrogen.

eter" provides a second. The magnetic thermometer measures the change in magnetic susceptibility of a sphere of chromic methylammonium alum suspended in the working space.

The He^3 refrigerator maintains an operating temperature in which a number of nuclei (Ho, Tb, U, Np) can be oriented to a significant degree. Therefore, a second refrigerator, similar to the prototype described above, has been constructed for use in the Bureau's nuclear research program. A modified working space, with aluminum "windows" to permit the passage of high-energy radiation, has been substituted for that containing the susceptibility detecting equipment. This refrigerator is portable, to permit movement between several particle accelerators. The ability to change temperatures rapidly will facilitate alternate measurements with oriented and unoriented nuclei.

[1] Continuously operating He^3 refrigerator for producing temperatures down to ¼°K, *by E. Ambler and R. B. Dove Review of Scientific Instruments* **32,** *737 (June 1961).*

Organizational Changes in NBS Weights and Measures Activity

WILLIAM S. BUSSEY has been named Assistant to the Director of the National Bureau of Standards for Weights and Measures Administration. He has been succeeded as Chief of the Office of Weights and Measures by Malcolm W. Jensen, formerly Assistant Chief.

In his new position, Mr. Bussey will be responsible for administrative advisory services to officials of the States on weights and measures matters. Mr. Bussey will serve as Chairman of the Weights and Measures Advisory Committee and also as Executive Secretary of the National Conference on Weights and Measures. He will develop recommendations dealing with model weights and measures laws and regulations and maintain a program of personal contact with weights and measures officials and organized weights and measures groups.

The Office of Weights and Measures, under Mr. Jensen, will provide technical services to the States and to business and industry in the area of measurement, specifically matters dealing with testing equipment, testing procedures, specifications, and tolerances for weighing and measuring devices; the design, construction, and use of standards of weight and measure and of instruments associated with such standards. The Office will participate in the training of State and local weights and measures officials in the technical aspects of their programs, and will undertake solution of various special measurement problems, provide liaison between State and local officials and Bureau scientific personnel, and participate in international activities concerning weights and measures standards and practical metrology.

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Growth Rate of Oxi

TO PROVIDE BASIC DATA on the mechanisms involved in the corrosion of metals, the Bureau is investigating the thin oxide films that sometimes form on metal surfaces during the corrosion process. In recent work, the thicknesses of such films were measured as they increased on four different crystallographic planes of copper single crystals immersed in oxygenated water. The measurements indicated that the type of oxide formed, its rate of growth, and its thermodynamic stability were greatly influenced by changes in oxygen concentration. The study, partially sponsored by the Corrosion Research Council, was made by Jerome Kruger of the Bureau's metallurgy laboratories.[1]

In an earlier Bureau study [2] of the chemical composition of the films formed on copper surfaces, it was found that both cuprous oxide and cupric oxide were produced. The first oxide formed as a vari-colored adherent layer on the metal surface and the second as a powdery outer layer. The present work was undertaken to obtain data on the rate of cuprous oxide formation, and to ascertain whether different oxygen concentrations affected film formation and stability under controlled laboratory conditions.

The copper single crystals used in the study were cylindrical in shape, approximately $\frac{5}{8}$ in. in diameter and 1 in. in length, with their axes parallel to the (110) crystallographic plane. This orientation permitted flat surfaces to be cut on the crystals parallel to the (110), (111), (100), and (311) planes.

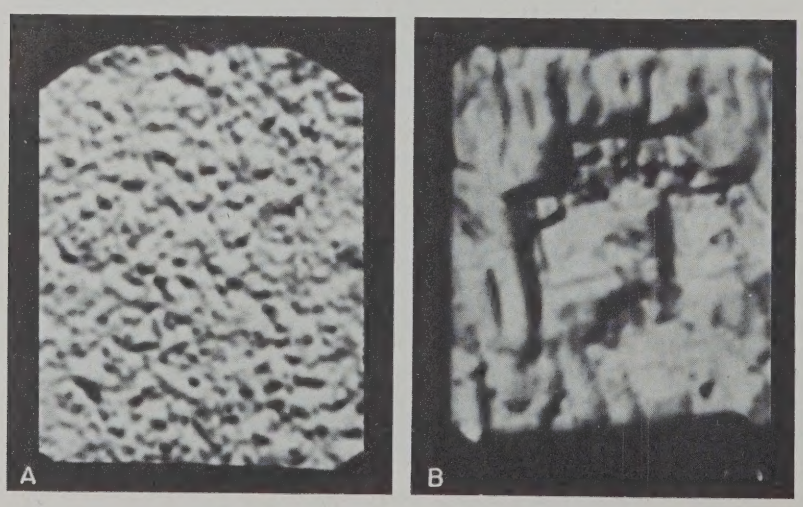
When a specimen prepared in this manner had been mounted on a glass stage, it was sealed in a closed all-glass apparatus. Two small iron bars were fitted into the glass stage so that the mounted specimen could be rotated magnetically about its axis to any desired position. Thus, as films formed on the various crystallographic planes during the oxidation process, they could be measured with a polarizing spectrometer. This instrument, also called an ellipsometer, measures thicknesses to ± 3 A or better by means of refractive index techniques.

In the first experiment with this equipment, a specimen was immersed in a solution of oxygen and pure water in equilibrium with pure oxygen at a pressure of 1 atm. Rapid oxidation in the form of cuprous oxide occurred initially on the specimen and then leveled off for a period of from 90 to 120 min on the different planes, after which cupric oxide started to form. Limiting thicknesses of the first coating ranged from 65 to 95 A on the four surfaces.

Spectrochemical analysis of the solution both before and after cupric oxide was observed showed the copper concentration in the solution to be less than 0.02 mg/liter in the first instance and 0.04 mg/liter in the second. This change in copper concentration indicated that cupric oxide precipitated on the cuprous oxide-copper surface when its solubility product constant was exceeded.

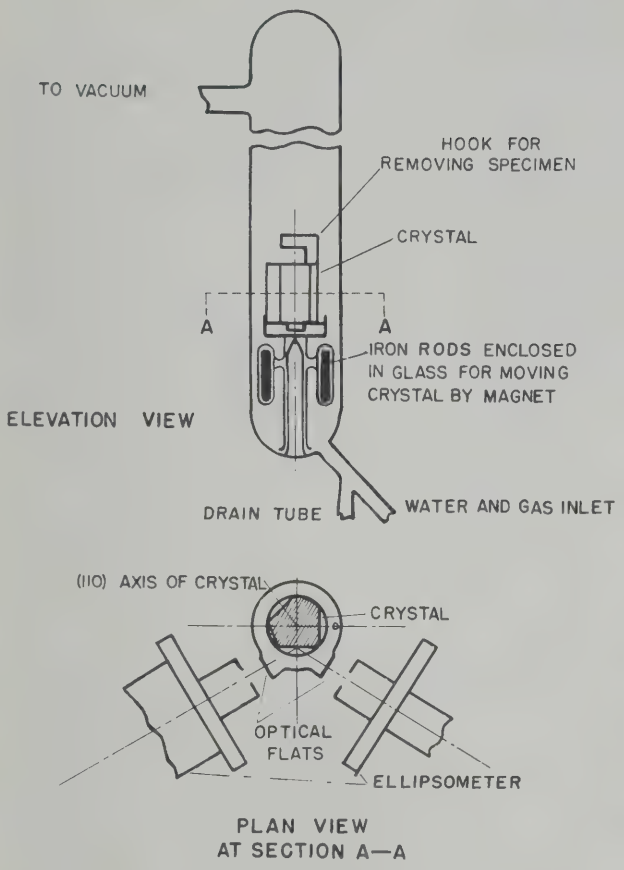
In the next experiment, the same environmental conditions were employed except that the liquid was stirred by bubbling in pure oxygen at increasing rates. Almost the same behavior was noted; however, with each increase in stirring rate a greater period of time elapsed before the first traces of cupric oxide appeared. This reaction tends to confirm the conclusions derived from the first experiment. Apparently the stirring carried the cupric ions away from the surface, with precipitation occurring in the body of the liquid. Thus additional time was required for cupric oxide to precipitate at the surface in an amount sufficient to affect the oxidation process.

Only 1 percent oxygen was used in the next experiment, with the total pressure brought up to 1 atm by the addition of helium. With this combination, the rate of cuprous oxide formation appeared to follow a



Electron micrographs of the (111) surface (A), and the (311) surface (B), of a copper single crystal after exposure to a 1 percent oxygen atmosphere. (× 21,500). These micrographs illustrate the discontinuous nature of the films formed on crystallographic planes with different orientations.

le Films on Copper



Apparatus used to study the formation of oxide films on metal surfaces. The four different surfaces cut parallel to the crystal axis are indicated in the plan view (bottom).

parabolic law, indicating that a diffusion process was controlling the growth rate. The film formed on the specimen exhibited rate constants of approximately 0.13 A^2/min on the (111) plane and 0.63 A^2/min on the other three planes. This marked difference in rate constants indicates that diffusion of copper ions through the film, rather than diffusion of oxygen in solution, controls the growth rate of corrosion products. A thickness of 160 A was attained at the end of 300 min; the measurements were then discontinued because a gradual change in refractive index took place. However, film thicknesses greater than those produced by the pure oxygen atmosphere formed on all planes.

When a 10-percent oxygen-helium atmosphere was used, lesser film thicknesses were found than in the 1-percent oxygen-helium mixture, but these thicknesses were always greater than those observed in the pure oxygen atmosphere. When the oxygen was increasd to 20 percent, the behavior approached that observed in the pure oxygen experiment, with cupric oxide appearing at the end of 240 min.

From the results of the study it appears that at low oxygen concentrations the cuprous oxide film is the stable phase, and that it grows to greater thicknesses than it does at high oxygen concentrations. Under the latter conditions, cupric oxide becomes the stable phase. The study showed that in both high and low concentrations, the (111) crystallographic plane exhibits the smallest rate of oxidation, possibly because it has the highest work function—that is, gives up its electrons less readily than do all of the other planes in the face-centered cubic space lattice of copper.

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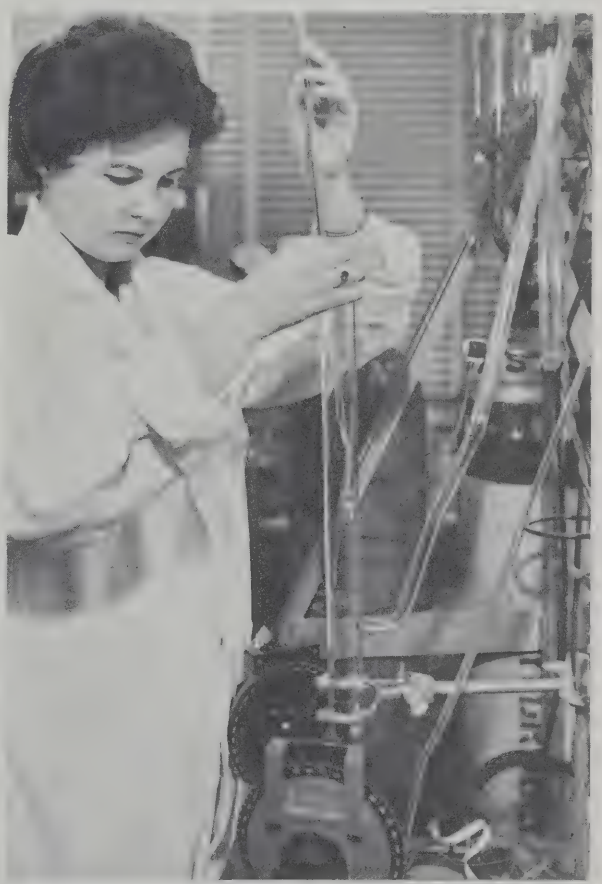
[1] *For further technical details, see* The oxide films formed on copper single crystal surfaces in water: II. Rate of growth at room temperature, *by Jerome Kruger, J. Electrochem. Soc.* **108,** *503 (1961).*

[2] The oxide films formed on copper single crystal surfaces in water: I. Nature of films formed at room temperature, *by Jerome Kruger, J. Electrochem. Soc.* **106,** *847 (1959)*; *also,* Corrosion Research, *NBS Tech. News Bull.* **43,** *150 (1959).*



Miss Joan Calvert inserts a specimen into the apparatus used to study the formation of oxide films on metal surfaces. Film thicknesses are measured with a polarizing spectrometer (center bottom).

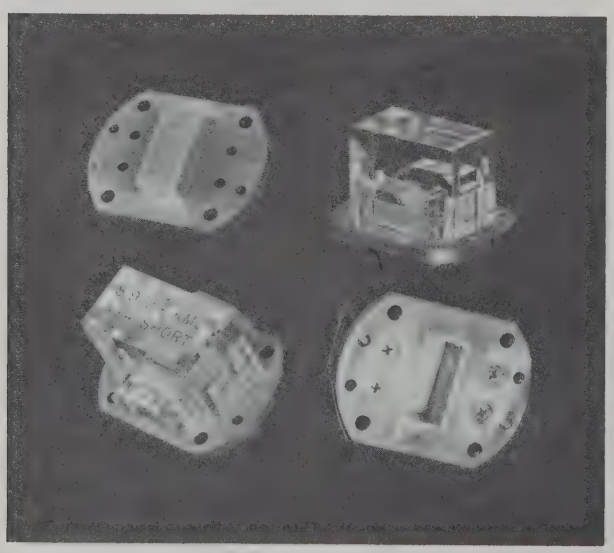
Microwave Impedance Calibration Service

Sixth of a Series on NBS Measurement and Calibration Services *

THE ELECTRONIC CALIBRATION CENTER of the Bureau Radio Standards Laboratory, Boulder, Colo., has recently initiated a service for the calibration of waveguide reflectors in the microwave frequency region. The service is presently offered in WR–90 waveguide size throughout the frequency range 8.2 to 12.4 Gc. Calibrations of high accuracy are obtained through the use of a modified reflectometer system. The waveguide reflectors are calibrated by comparison with an NBS-designed working standard employing a straightforward method that can be adapted for use in other standards laboratories. The service is currently being utilized by both government and industrial standards laboratories. Calibration of these devices will provide standards of comparison for calibrating various types of microwave impedance measuring systems.

Impedance in microwave circuits is often not uniquely defined. However, the behavior of propagating waves can be determined in terms of two waves traveling in opposite directions. A wave incident upon an arbitrary change in an otherwise uniform transmission line results in a reflected wave, the relative magnitude and phase of which are determined by the nature of the change. The ratio of the reflected wave to the incident wave at a given reference plane is termed the reflection coefficient. The two opposite-traveling waves also interfere with each other to form a standing-wave pattern. For voltage waves, the ratio of the maximum amplitude to minimum amplitude of the standing wave is called the voltage-standing-wave-ratio (VSWR) and is linearly related to the voltage reflection coefficient magnitude. The calibration system in use at the Bureau is designed to measure accurately the magnitude of the voltage reflection coefficient for the dominant mode of propagation.

Precision quarter-wavelength short circuit standards against which interlaboratory standard reflectors are calibrated.



Devices designed to effect a standard value of reflection coefficient are termed "reflectors." They are sometimes called standard reflections or standard mismatches. Reflectors currently being manufactured for rectangular waveguide are generally of the fixed type, and a separate reflector is needed for each value of reflection coefficient. The reflection is usually accomplished by a change in one of the inside transverse dimensions of the waveguide at the plane of the mounting flange. In most such reflectors, the smaller dimension of the waveguide is made to be less than the same dimension for standard size waveguide. The reflector contains a low-reflection absorptive element provided with means for sliding it along the axis of the waveguide to permit averaging out the reflected signal from the absorptive element. A typical set of waveguide reflectors for a given waveguide size consists of 5 reflectors with nominal VSWR's of 1.05, 1.10, 1.20, 1.35, and 1.50. Corresponding reflection coefficient magnitudes are approximately 0.025, 0.05, 0.09, 0.15, and 0.20.

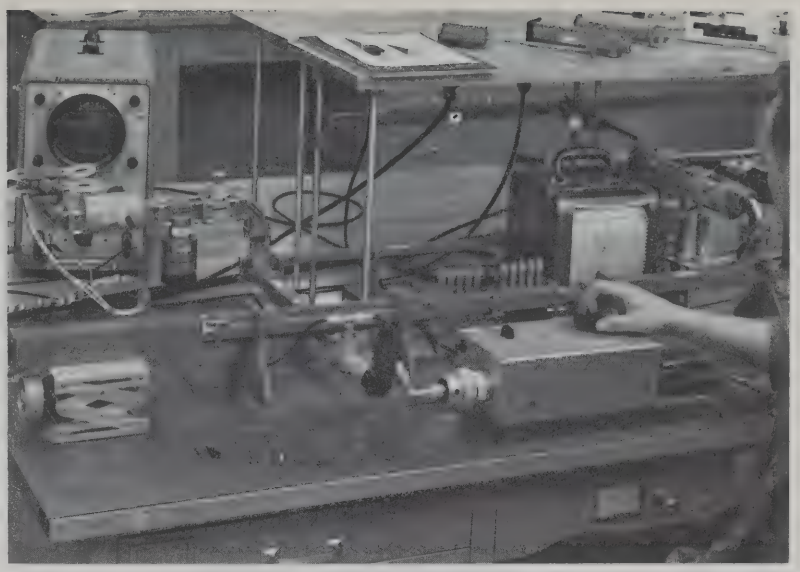
NBS microwave impedance calibration service in WR-90 wave guide

Nominal value		Accuracy of calibration
VSWR	Reflection coefficient magnitude ($\|\Gamma\|$)	Percent of ($\|\Gamma\|$)
1.05	0.025	1.5
1.10	.05	1.5
1.20	.09	1.0
1.35	.15	1.0
1.50	.20	1.0

Many standards laboratories maintain a set of reflectors for each of the common waveguide sizes. These reflectors are used to calibrate microwave impedance measuring instruments such as slotted lines and reflectometers. Reflectors calibrated by the Bureau should be employed to calibrate working sets of reflectors.

The surface of the waveguide flange of a reflector submitted to the Bureau for calibration is inspected for flatness and freedom from nicks and scratches. Since the reflector must be attached to a precision waveguide section incorporated in the calibration equipment, such defects might damage the precision section and are cause for rejection. If the mounting flange surface of the reflector is suitable for connection to the calibration equipment, it is checked electrically for variations in the reflection coefficient due to the following:

**Previous articles have been:* Calibration of Gage Blocks, *Feb. 1961;* Thermocouple Calibrations, *Mar. 1961;* Calibration of Platinum Resistance Thermometers, *Apr. 1961;* Calibration of Inductive Voltage Dividers, *May 1961;* and X- and Gamma-Ray Calibration, *July 1961.*



Adjusting a precision variable attenuator used to calibrate interlaboratory standard reflectors against NBS working standards. An interlaboratory standard is supported by the laboratory jack at lower left.

1. Rotational movement of the sliding termination.
2. Transverse movement of the sliding termination.
3. Variations in successive maximums and/or successive minimums obtained with the sliding termination.
4. Instability in positioning the sliding termination for a maximum or a minimum.
5. Variation in reflection coefficient between the two possible mounting positions of the reflector.

Reflectors exhibiting excessive variations in any of the above usually are not calibrated.

By use of a modified reflectometer system described by G. F. Engen and R. W. Beatty [1] and placed in operation by E. Campbell, the reflection coefficient magnitude is determined with reference to the NBS standard.[2] The maximum and minimum reflection coefficient magnitudes are measured and the two results averaged to obtain the magnitude of the reflection coefficient due to the discontinuity at the flange. The NBS working standards currently used are electroformed silver quarter-wave short circuits, the reflection coefficient magnitudes of which have been evaluated to an accuracy of ±.05 percent.

[1] Microwave reflectometer techniques, *by G. F. Engen and R. W. Beatty, IRE Trans. on Microwave Theory and Techniques, MTT–7, No. 3, 351 (July 1959).*

[2] Recently developed microwave impedance standards and methods of measurement, by *R. W. Beatty and D. M. Kerns, IRE Trans. on Instr., I–7, Nos. 3 and 4, 319 (Dec. 1958).*

Separation of Hafnium From Zirconium

HAFNIUM AND ZIRCONIUM, ordinarily very difficult to distinguish chemically, can now be separated by a single-step anion-exchange procedure [1] developed by the Bureau. The separation for analysis is obtained by using a strong quaternary-amine anion-exchange resin column with diluted sulfuric acid as eluting solution. After separation of a mixture containing approximately 100 mg each of hafnium and zirconium, a spectrochemical examination showed only a few ppm (parts per million) cross contamination.

The separation procedure was developed by J. L. Hague and L. A. Machlan as part of a continuing program to develop accurate methods for determining the composition of materials. In many uses, hafnium and zirconium can be considered as one constituent. However, in nuclear reactors and certain other applications, the difference in their nuclear properties makes it important to know the relative amounts of each present. A practical chemical procedure has not been available to determine the relative amounts of hafnium and zirconium present or to follow the commercial purification of these two elements. The procedure developed is particularly useful for the determination of zirconium in hafnium-base materials.

To find the eluting solutions that would give the best separation, a systematic survey of the elution of hafnium and zirconium in diluted sulfuric acid, and in solutions of hydrochloric and sulfuric acids in water, was conducted. The most satisfactory separation was obtained by using 3.5 percent sulfuric acid solution.

The sample is prepared for the separation by dissolving it in a sulfuric-hydrofluoric acid solution and then fuming off the hydrogen fluoride. Polystyrene columns, loaded with a commercial anion-exchange resin, are conditioned with 3.5 percent sulfuric acid solution. After the diluted sample solution is added to the column, the hafnium fraction and then the zirconium fraction are eluted with 3.5 percent sulfuric acid solution. The hafnium and zirconium, in separate fractions, are then precipitated with cupferron, filtered, ignited, and weighed as the oxide.

[1] *For further information, see* Separation of hafnium from zirconium by anion exchange, *J. L. Hague and L. A. Machlan, J. Research NBS* **65A** *(Phys. and Chem.), No. 1, 75 (Jan.–Feb. 1961).*

Survey of Methods for Measuring the Pentosans and Carboxyl Content of Cellulose

THE BUREAU has made a survey of the principal methods used in this country and abroad for the determination of both pentosans and carboxyl in cellulose. Laboratories in Austria, Canada, Finland, Germany, and Sweden joined with U.S. laboratories in providing experimental data for the study, conducted by W. K. Wilson and John Mandel of the Bureau staff. From a statistical analysis of the data it was possible to arrive at a standard method for the determination of pentosans in cellulose, and to select tentative standard methods for determining carboxyl content.[1]

Minor functional groups in cellulose, such as carboxyl and pentosans, influence the processing of the fiber into the various materials for which it is used. Hence, a number of analytical methods have been developed to measure their content. However, the degrees of precision and reproducibility that can be achieved with these methods can be found only from a comparison of interlaboratory results. The present study was therefore undertaken at the request of the International Committee for Cellulose Analysis (ICCA) and other technical groups which are trying to improve methods of chemical analysis.

Determination of Pentosan Content

In the pentosan study, the orcinol colorimetric method employed in Sweden was compared with the aniline acetate colorimetric and bromination methods widely used in this country. Specimens from eight different samples of ICCA pulp were supplied to seven laboratories, together with detailed instructions for the application of each method. In general, three separate tests were run for each group of specimens, according to each of the three methods, in each laboratory. The data obtained from these tests were then evaluated.

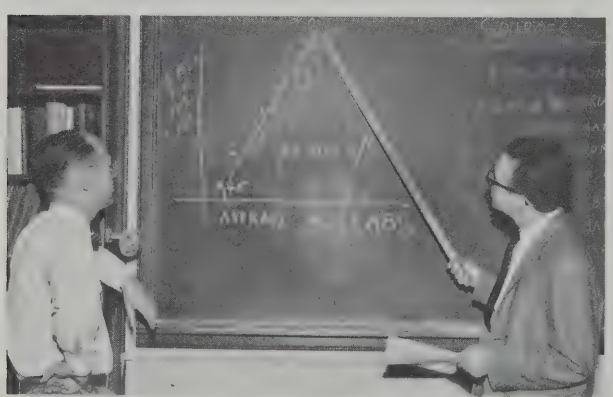
A mathematical "linear model" [2] previously developed for analyzing the results of interlaboratory testing was applied to these data, and the orcinol method was found to be superior to the aniline acetate method. About five measurements by the latter method were necessary to achieve the same precision as one measurement by the orcinol method. The values obtained by both methods compared favorably with those obtained by the bromination method, which is used mainly to determine larger amounts of pentosans.

Determination of Carboxyl Content

The amount of carboxyl in cellulose is generally found either by determining the acid liberated upon addition of de-ashed pulp to a salt solution, or by determining the decrease in cation concentration of a salt solution by de-ashed pulp. Several different reaction procedures have been developed and a total of eight methods were used in this study. A different method was used by each of the eight participating laboratories on each of eight different ICCA pulps, and in addition, four of the methods were tested in two or more laboratories.

From the 15 sets of data thus obtained, it was found that the major source of within-laboratory variability arose from differences in the manner in which different materials responded to the particular technique used. Results obtained on the same group of specimens by two or more methods in the same laboratory varied more than did results achieved with the same method in different laboratories. The coefficient of variation ranged from about 20 percent for pulps with 0.5 mM of carboxyl per 100 g of specimen to about 5 percent for pulps containing about 5 mM of carboxyl per 100 g. The application of the linear model to the data showed that the use of a standard reference material in determining carboxyl content would materially improve interlaboratory precision.

John Mandel and W. K. Wilson discuss the linear model used in analyzing the results of interlaboratory evaluations. By means of this technique, distinctions can be made between random and systematic sources of error in measuring the chemical properties of materials.



[1] *For further technical details, see* Determination of pentosans, *by W. K. Wilson and John Mandel, TAPPI* **43,** *998 (1960)*; *also* Determination of carboxyl in cellulose, *by the same authors, TAPPI* **44,** *131 (1961)*.

[2] The measuring process, *by John Mandel, Technometrics* **1,** *251 (1959)*; The interlaboratory evaluation of testing methods, *by John Mandel and T. W. Lashof, ASTM Bull.* **239,** *53 (1959)*; *and* Intercomparison of laboratory test methods, *NBS Tech. News Bull.* **44,** *166 (1960)*.

Fast Electron Dose From a Narrow Beam Source

THE BUREAU has made a calculation of the penetration in air of fast electrons from a beam source. The work [1] was sponsored by the Atomic Energy Commission and was part of a program to develop methods for calculating the dose from a source geometry involving two spatial variables. Possible applications of such calculations include the determination of the spatial distribution of the dose in therapy involving electron beams and the estimation of the resolving power of large single crystals in high-energy radiography.

The experimental situation is that of a collimated beam of monoenergetic electrons originating at a point in the medium. As the electrons move about, their energy is dissipated. The basic quantity to be determined is the energy dissipated in a small volume located at specified distances from the source point and the center line of the initial beam.

Calculations of the energy dissipation distributions for fast electrons in infinite and homogenous media have been reported for the simplest source geometries.[2, 3] The present calculation represents an extension of the earlier work and is a special application of the moment-fitting techniques developed at the Bureau and applied initially to problems of X-ray penetration.[4, 5]

[1] *For further information, see* Calculated energy dissipation distribution in air by fast electrons from a gun source, *by John E. Crew, J. Research NBS* **65A** *(Phys. and Chem.), No. 2, 113 (1961).*

[2] *L. V. Spencer. Phys. Rev.* **98**, *1597 (1955).*

[3] *L. V. Spencer, NBS Mono. 1 (1959). For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. Price 45 cents.*

[4] *L. V. Spencer and U. Fano, J. Research NBS* **46**, *446 (1951).*

[5] *Fano, Spencer, and Berger, Encyclopedia of Physics (Springer-Verlag, Berlin) 38, Part 2 (1959).*

Rate of the Reaction NO+N

RESEARCH conducted by John Herron [1] of the Bureau has shown that the rate constant of the reaction $NO+N \rightarrow N_2+O$ is $1.0\pm0.5\times10^{13}$ cm^3/mole/sec at room temperature. This reaction—one of the fastest second-order reactions known—occurs naturally in the upper atmosphere, and may contribute to production of the airglow. The Advanced Research Projects Agency supported the investigation, which was conducted to provide basic information concerning rate-constant data of nitrogen atom reactions.

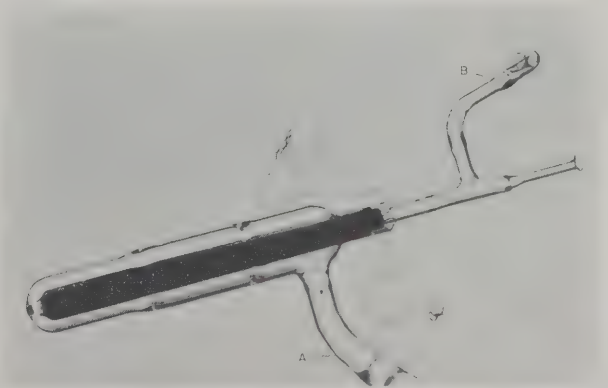
In this investigation, active nitrogen (N), produced by exposing $N_2{}^{14}$ to 2450-Mc electrodeless discharge, was reacted with nitric oxide ($N^{15}O$) in a special glass reaction vessel. These gases entered the vessel through separate glass tubes, and all gases were exhausted by a vacuum system. By varying the flow rates, and the length of the reaction zone, the reaction time could be varied from 0.5×10^{-3} to 1.7×10^{-3} sec.

A fraction of the products of the reaction between the gases were leaked through a 0.025-mm hole in the bottom of the reaction vessel to a mass spectrometer for analysis. The inlet system and ion source of the spectrometer were designed so that a fraction of the gas could be ionized without undergoing collision with the walls. From the known concentrations of reactants and products, and the reaction time, the rate constant k of the reaction was calculated. The value of k, from a series of six determinations (all at room temperature), was found to be $1.0\pm0.5\times10^{13}$ cm^3/mole/sec.

In addition to the N_2 ($N^{14}N^{15}$) and O found in the reaction products, small amounts of $N^{14}O$ and O_2 were also detected. From the rates of the reactions giving rise to these products, it seems almost certain that they were formed by reactions taking place on the walls of the mass spectrometer ion source.

Further work on nitrogen reaction rates is being planned. The next reaction to be investigated is that between nitrogen and ozone. This reaction also occurs in the upper atmosphere.

[1] *For further details, see* Rate of the reaction NO+N, and some heterogeneous reactions observed in the ion source of a mass spectrometer, *by John T. Herron, J. Research NBS, Sec. A (Phys. and Chem.) to be published.*



Reaction vessel used in the study of the rate of the reaction $NO+N \rightarrow N_2+O$. Active nitrogen, produced by exposing nitrogen gas to an electrodeless discharge, enters the vessel through tube A, and mixes with $N^{15}O$ coming down a second center tube (not shown). The gases, and the products of their reaction, are exhausted by a vacuum pump through tube B. A small amount of the gas is "leaked" through a 0.025-mm hole in the bottom to a mass spectrometer for analysis. Results indicate that the rate of this reaction, one of the fastest second-order reactions known, is $1.0\pm0.5\times10^{13}$ cm^3/mole/sec at room temperature.

Pipe Sizes for Plumbing Stacks in Buildings

A STUDY at the Bureau on the mechanics of flow in main vertical plumbing drains and vents in buildings has provided information useful in preventing oversizing of pipes.

The objective has been to develop criteria which would provide pipes adequate to carry maximum expected loads, but not unnecessarily large and costly. The investigation has resulted in a number of equations, tables, and charts to compute pipe dimensions, and provide drains and vents ample but not excessive in size. The study [1] was conducted by Robert S. Wyly and Herbert N. Eaton. Their program included both laboratory work at the Bureau and compilation and evaluation of data from other sources.

Lack of adequate knowledge of hydraulics and pneumatics of building drainage systems is a serious handicap to writers of plumbing codes and design handbooks. This lack of knowledge has resulted in a great diversity in code requirements. For example, a review of a number of municipal plumbing codes showed requirements differing by as much as a factor of nine in fixture unit loadings for drainage stacks.

Information obtained in the study supports some of the technical requirements of recent plumbing codes patterned after the American Standard National Plumbing Code ASA A40.8–1955, the development of which was aided by earlier Bureau research.

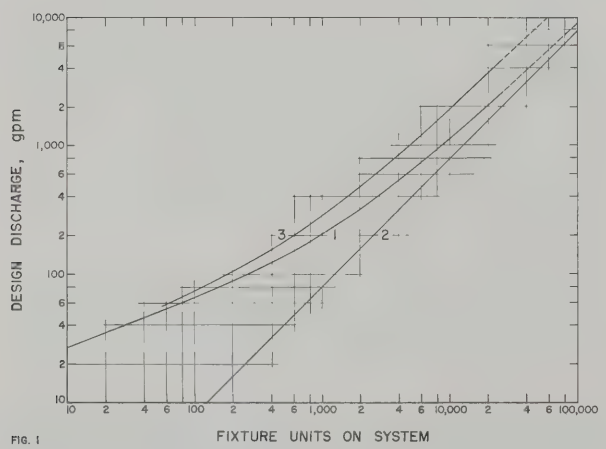


Figure 1. Curve 1 makes possible an estimate of the peak discharge for design purposes (design flow) in gallons per minute in building drainage systems having "fixture unit" loads totaling between 10 and 100,000. The "fixture-unit" weight for a particular plumbing fixture expresses its relative load-producing effect. Curve 2 represents the average discharge from a system of water closets, each of which discharges 4 gal of water once in 5 min. The ordinates of curve 3 were obtained by addition of the ordinates of curves 1 and 2 for each abscissa value.

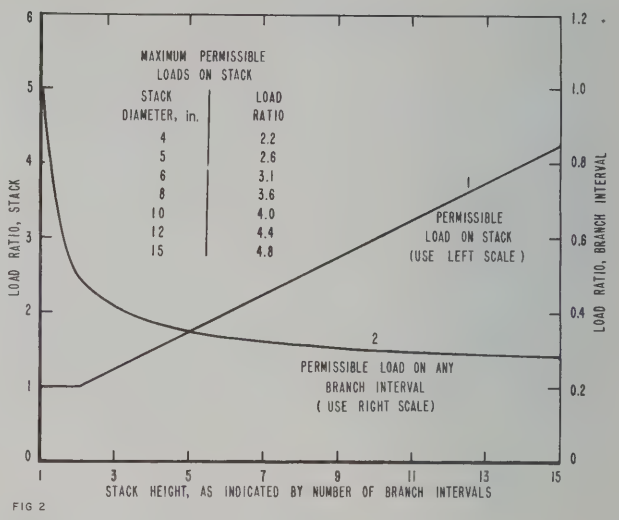


Figure 2. Permissible loadings, for soil and waste stacks of various heights, as developed from studies of flow in vertical plumbing drains in buildings. The load ratio is the ratio of the load under consideration (in fixture units) to the load (in fixture units) permitted on a stack having one branch interval. If total stack loadings are increased in accordance with curve 1, individual branch interval loadings must be decreased in accordance with curve 2. Inset shows maximum permissible loads on stacks of various diameters.

Two basic factors determine pipe size requirements: (1) the flow capacity of the pipe under service conditions; and (2) the design flow expected under service conditions.

The design flow is the volume rate of flow estimated to occur sufficiently often to cause unsatisfactory performance if not adequately provided for in the design of the system. For systems serving more than a very limited number of fixtures, this requires a systematic method of computing the simultaneous discharge rate from a given group of fixtures which, over a long period of time, will not be exceeded except for a specified small fraction of time during periods of heavy use.

The design flow is computed by a method based on the theory of probability.[2, 3] First of all, the total of "fixture units" to be installed in the building or on the pipe under consideration is calculated. Each type of fixture has a fixture unit value or "weight," which depends on the rate of discharge from the fixture, as well as on the duration of a typical operation, and the average time between successive operations during heavy usage. Thus by the fixture unit method of rating, each fixture is rated on its probable load-producing effect.

The present study provides a curve (curve 1 of fig. 1) for estimating the peak discharge for design purposes (design flow) in building drainage systems having fixture units totaling between 10 and 100,000. The concepts on which the curves in this figure are based were developed in earlier studies at the Bureau and elsewhere; and a curve similar to curve 1, covering fixture unit totals between 10 and 3000, was developed in an earlier NBS study.[2] In the current study, for fixture unit loads up to 4800, curve 1 has been computed from tables of the cumulative binomial probability distribution. The portion of the curve beyond 4800 fixture units has been computed from Poisson's approximation to the binomial distribution. Curve 2 of figure 1 represents the average discharge from a system of water closets, each of which discharges 4 gal. of water once in 5 min. The ordinates of curve 3 were obtained by addition of the ordinates of curves 1 and 2 for each abscissa value.

The current investigation also resulted in calculation of permissible loadings for soil and waste stacks of various heights (table and fig. 2). Load limits for stacks and branch intervals in multistory systems may be determined by multiplying the table values (for one-interval stacks) by the appropriate load ratio (for multi-interval stacks) from figure 2. In addition, permissible lengths of vent stacks for various loads on soil and waste stacks were calculated.

When water is discharged into a vertical drain (soil or waste stack) from a horizontal drain, it accelerates rapidly under the influence of gravity, tending to attain an annular cross section and to reach practically its terminal velocity in one or two stories' height of fall. An equation [1, 4] was developed for computing this terminal velocity. Another calculation showed the height of fall (terminal length) at which 99 percent of computed terminal velocity would occur.

When water falls in a soil or waste stack, it carries air along with it, enclosed for the most part within a central "core." This leads to substantial fluctuations of pneumatic pressure within the system. If suitable provisions are not made to relieve these pneumatic disturbances, the water seals in the fixture traps will not provide satisfactory protection against the passage into the building of objectionable and possibly dangerous sewer gases. Another objection to a poorly vented system is that drainage in such a system may be particularly noisy. In order to design the venting system properly, it is necessary to estimate the rate of air flow which must be carried for any given rate of water flow. Equations were developed to make this estimate for the vertical mains of the venting system.[1, 5]

When water is being carried in a drainage stack simultaneously from above and from a connecting horizontal branch, hydrodynamic back pressure is generated within the horizontal branch. This may affect the capacities of horizontal branches.

When one or more horizontal branches discharge large quantities of water into a soil or waste stack at a given elevation, without any discharge from higher elevations, energy adjustments at the junction may cause the stack to fill.[6] Experiments have provided data from which a reasonable upper limit on rate of discharge can be set to avoid the generation of too great pressures at the junction.

Maximum loads for soil and waste stacks having one branch interval

Stack diameter	Maximum load
in.	*fixture units*
2 (waste only)	8.
2½ (waste only)	20.
3 (soil)	*30.
3 (waste)	48.
4 (soil or waste)	240.
5 (soil or waste)	540.
6 (soil or waste)	930.
8 (soil or waste)	2, 100.
10 (soil or waste)	3, 750.
12 (soil or waste)	5, 850.
15 (soil or waste)	10, 500.

**Not more than 2 water closets.*

[1] Capacities of stacks in sanitary drainage systems for buildings, *by Robert S. Wyly and Herbert N. Eaton, National Bureau of Standards Monograph 31 (1961), available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. (35 cents).*

[2] *See* "Methods of Estimating Loads in Plumbing Systems," *by Roy B. Hunter, NBS Building Materials and Structures Report* **BMS65** *(1940).*

[3] Fixture unit ratings as used in plumbing system design, *Housing Research Paper 15, Housing and Home Finance Agency (1951).*

[4] *See also* "Report on Hydraulics and Pneumatics of Plumbing Draining Systems—I," *by F. M. Dawson and A. A. Kalinske, University of Iowa Studies in Engineering, Bulletin 10 (1937).*

[5] *See also* "Report on the Hydraulics and Pneumatics of the Plumbing Drainage System," *by F. M. Dawson and A. A. Kalinske, Technical Bulletin 2 of the National Association of Master Plumbers of the United States, Inc. (1939).*

[6] *See* "Recommended minimum requirements for plumbing in dwellings and similar buildings," *(1924), NBS Building and Housing Publication* **BH2**; *also* "Recommended minimum requirements for plumbing," *(1932), NBS Building and Housing Publication* **BH13**, *available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. (price $1.50).*

46th National Conference on Weights and Measures

June 12–16, 1961

Nearly 400 delegates attended the 46th National Conference on Weights and Measures (June 12–16) in Washington, D.C., with weights and measures officials representing 35 States, the District of Columbia, and the Commonwealth of Puerto Rico. Also represented were the Institute of Weights and Measures Administration of Great Britain, and the Standards Division of the Department of Trade and Commerce of Canada.

Several resolutions were among the principal actions of the Conference, which is sponsored by the National Bureau of Standards in cooperation with weights and measures officials throughout the United States. One resolution commended President Kennedy for his intention to establish a consumer counsel in the President's office, and offered the full cooperation of the National Conference on Weights and Measures. Another resolution urged that manufacturers, packers, and advertising agencies study and survey possible areas of deceptive packaging and labeling to assure adherence to existing laws, regulations, and moral obligations.

Speaking at the opening session, Secretary of Commerce Luther H. Hodges cited the long, successful relationship of NBS and the National Conference, and their mutual efforts to assure "that equity may prevail" in the Nation's commercial transactions. The Secretary noted the increased efforts of the Bureau to extend and refine the standards of physical measurement, citing the importance of such efforts to the country's economic progress, to its military and space programs, as well as to the advancement of science itself.

Referring to the wide technical activities of the Department of Commerce, Secretary Hodges said, "We have asked the Congress to authorize a new Assistant Secretary of Commerce for Science and Technology to help us make the most intelligent use of Commerce's various technical agencies, and to work with other Government agencies and private industry to achieve more fully the benefits that can be derived from an estimated $14 billion now being spent annually for research and development. We believe Congress will grant our request."

An address by Senator Philip A. Hart of Michigan highlighted the Conference luncheon on June 14. The Senator discussed the hearings which the Senate Antitrust and Monopoly Subcommittee is conducting on deceptive practices in packaging and labeling. Senator Hart declared that such practices could threaten the very preservation of our free enterprise economy.

NBS Director A. V. Astin, President of the National Conference, addressing the Conference on June 13, reviewed recent technical achievements of the Bureau in the field of measurement, giving particular attention to accomplishments of significance to the weights and measures field.

Conference speeches on package standardization, package labeling, weights of frozen turkeys, bread tolerances, and quantity control in a large industry dealt with various aspects of the question of ethical packaging and labeling. A technical paper on progress in measurement of moisture in grain, two addresses on phases of the weights and measures programs in other countries, and a report from the Secretary of the National Conference rounded out the program.

One highlight of the Conference was an address by J. R. Roberts, Chief Inspector of Weights and Measures, Manchester, England, and Secretary, The [British] Institute of Weights and Measures Administration. Mr. Roberts described the apprenticeship, training and education, and examination which lead to the certification and status of a weights and measures official in Great Britain.

Food and Drug Commissioner G. P. Larrick, U.S. Department of Health, Education, and Welfare, discussed the public confusion caused by some current practices in packaging and labeling. He offered to deputize as agents of the Food and Drug Administration State officials interested in investigating possible violations in goods originating outside their own States. Commissioner Larrick also promised help in bringing legal action where indicated.

R. W. Coyne, President of the Distilled Spirits Institute, Inc., spoke on the unusual degree of self-discipline that his industry has imposed upon itself in its relations with its customers. This, said Mr. Coyne, contrasts sharply with many current packaging and labeling practices and with the resultant customer confusion.

J. L. Littlefield, Chief of the Foods and Standards Division, Michigan Department of Agriculture, also discussed the growing tendency toward packaging, labeling, and advertising to confuse rather than to inform. Mr. Littlefield urged the Conference to reiterate its stand against misleading terms in advertising and labeling.

J. H. Lewis, Chief, Weights and Measures Section, Department of Agriculture, State of Washington, discussed the difficulty and complexity of attempts to arrive at standards for the amount of water that can ethically be frozen into turkeys.

William Schieb, Maintenance Instructor, American Institute of Baking, Chicago, Ill., reported on a study by the baking industry into reasonable weight variations in packaged bread, thus giving the National Con-

ference on Weights and Measures an appropriate basis for moving toward more realistic weight tolerances in the future.

J. H. Chaloud, Associate Director, Product Development Division, The Procter & Gamble Co., described the efforts exerted, and the methods used, by his organization to maintain product quantity. According to Mr. Chaloud, his company believes in working with all weights and measures regulatory groups as the surest way of obtaining the mutual ultimate aim of giving a full measure of service to the customer.

Of great technical interest was a paper presented by W. W. Walton, Chief of the NBS Organic Building Materials Section. Dr. Walton reported on a research and development project of the Bureau that is designed to answer certain basic questions with respect to the moisture in grain and to develop portable equipment that will afford accurate measurement of this moisture.

A. E. Diaz, Head of the Weights and Measures Division, Economics Stabilization Administration, Commonwealth of Puerto Rico, reported on a one-year Central American study which he recently concluded as a representative of the Technical Assistance Program of the United Nations. The purpose of the study was to set up weights and measures control programs in Central America on national bases, and to encourage the establishment of a Central American Research Institute for Industry at Guatemala City, which would supply standards and technical assistance to the national bureaus. The standardization of weights and measures in Central America has been considered of paramount importance as part of the preparations for the Common Market, under the Economic Integration Program.

W. S. Bussey of NBS, reporting as Secretary of the National Conference, summarized weights and measures activities of the States, counties, and cities, and at the National Bureau of Standards. Complete new weights and measures laws were enacted during the past year by Alaska and Tennessee, Mr. Bussey reported, and numerous amendments to standing laws were enacted by several other States. The technical training for weights and measures officials, conducted by the NBS Office of Weights and Measures, was described.

The Conference, which was organized in 1905, is sponsored by the National Bureau of Standards and brings together weights and measures officials, as well as representatives of business and industry. Model laws, specifications, tolerances, regulations, and enforcement practices are recommended by the Conference for adoption by the various States, which have the legal responsibility of regulating commercial weighing and measuring devices, and of controlling commercial transactions involving quantity. The Bureau, through its Office of Weights and Measures, cooperates with the States in this endeavor by providing reference standards, calibrating services, and a wide range of technical advisory programs. Thus, the national standards of length and mass, which are in the custody of the Bureau, are translated into everyday use.

Officers elected for the ensuing year were: Robert Williams of New York, Chairman; Paul DeVries, New Jersey, H. H. Houston, Colorado, F. F. Thompson, Louisiana, and D. M. Turnbull, State of Washington, Vice Chairmen; and C. C. Morgan, Indiana, Treasurer. Dr. A. V. Astin, National Bureau of Standards, will continue to serve as President of the Conference; and W. S. Bussey, Assistant to the NBS Director for Weights and Measures Administration, will serve as Executive Secretary of the Conference.

Publications of the National Bureau of Standards

Periodicals

Technical News Bulletin, Vol. **45,** No. 7, July 1961. 15 cents. Annual subscription: $1.50, 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Basic Radio Propagation Predictions for October 1961. Three months in advance. CRPL–203, issued July 1961. 15 cents. Annual subscription: $1.50, 50 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75.

Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75.

Section D. Radio Propagation. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75.

Section A. Physics and Chemistry, Vol. **65A,** No. 4, July–August 1961. 70 cents.

Electrical properties and kinetics of electrode reactions, R. J. Brodd.

Effect of hydrostatic pressure upon the relaxation of birefrigence in amorphous solids, R. M. Waxler and L. H. Adams.

Vapor pressures of platinum, iridium, and rhodium, R. F. Hampson, Jr., and R. F. Walker.

Crystallization of bulk polymers with chain folding: theory of growth of lamellar spherulites, J. D. Hoffman and J. I. Lauritzen, Jr.

Phase equilibrium relations in the binary system barium oxide-niobium pentoxide, R. S. Roth and J. L. Waring.

Solid state reactions involving oxides of trivalent cations, S. J. Schneider, R. S. Roth, and J. L. Waring.

Gamma irradiation of fluorocarbon polymers, R. E. Florin and L. A. Wall.

Inhibition of diffusion flames of methyl bromide and trifluoromethyl bromide applied to the fuel and oxygen sides of the reaction zone, E. C. Creitz.

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Publications (Continued)

Section D. Radio Propagation, Vol. **65D,** No. 4, July–August 1961. 70 cents.

Almost fifty years of URSI, J. H. Dellinger.

Power density requirements for airglow excitation by gyro-waves, V. A. Bailey.

On the validity of some approximations to the Appleton-Hartree formula, K. Davies and G. A. M. King.

Amplitude and angular scintillations of the radio source Cygnus–A observed at Boulder, Colo., R. S. Lawrence, J. L. Jespersen, and R. C. Lamb.

Digital methods for the extraction of phase and amplitude information from a modulated signal, R. S. Lawrence, J. L. Jespersen, and R. C. Lamb.

Comparison between mode theory and ray theory of VLF propagation, H. Volland.

Antenna coupling error in direction finders, C. W. Harrison, Jr.

The electrically short antenna as a probe for measuring free electron densities and collision frequencies in an ionized region, R. W. P. King, C. W. Harrison, Jr., and D. H. Denton, Jr.

Effect of multiple atmospheric inversions on tropospheric radio propagation, F. H. Northover.

A few observations of the perturbations in the phase of the low frequency ground wave, J. M. Ross and J. E. Kirch.

Smooth earth diffraction calculations for horizontal polarization, L. E. Vogler.

On the theory of mixed-path ground-wave propagation on a spherical earth, J. R. Wait.

Technical Note

The following Technical Note is available from the Office of Technical Services, U.S. Department of Commerce, Washington 25, D.C. (Order by PB number).

Bibliography on ionospheric propagation of radio waves (1923–1960), W. Nupen, NBS TN84 (PB161585) (1961) $7.00.

Publications in Other Journals

The glass electrode, R. G. Bates, Book, Reference Electrodes, edited by D. J. G. Ives and G. J. Janz (Academic Press, Inc., New York, N.Y.), Chapter 5, 231–269 (1961).

A recalibration of the NBS standard thermal neutron flux, E. R. Mosburg, Jr., and W. M. Murphey, J. Nuclear Energy (Reactor Science and Technology Pt.) **14,** 25–30 (1961).

The relationship of low-height ionosonde echoes to Auroral-zone absorption and VHF *D*-scatter, J. K. Olesen and J. W. Wright, J. Geophys. Research **66,** No. 4, 1127–1134 (Apr. 1961).

Trends in the technology of automatic data processing, S. N. Alexander, AMA Report **43,** 38–42 (1960).

Use of the bulge test for determining mechanical properties of stainless steel foil, D. B. Ballard, Materials Research and Standards **1,** No. 6, 471–473 (June 1961).

Some comments on the use and preparation of hard gallium alloys, G. G. Harman, Rev. Sci. Instr. **32,** No. 4, 465–466 (April 1961).

On the nature of equatorial spread-*F,* R. Cohen and K. L. Bowles, J. Geophys. Research **66,** No. 4, 1081–1106 (Apr. 1961).

A study of the chemical reactions in kilning bone char, F. G. Carpenter and V. R. Deitz, Proc. 6th Tech. Session on Bone Char 1959, p. 321 (Bone Char Research Project Inc., Charlestown, Mass., 1961).

Rate of vaporization of refractory substances, R. F. Walker, J. Efimenkof, and N. L. Lofgren, Book, Physical Chemistry in Aerodynamics and Space Flight (Pergamon Press, Inc., London, England), p. 24–30 (1961).

Analysis of pyrolyzates of polystyrene and poly(methyl methacrylate) by gas chromatography, F. A. Lehmann and G. M. Brauer, Anal. Chem. **33,** No. 6, 673–676 (May 1961).

A note on phase velocity of VLF radio waves, J. R. Wait and K. P. Spies, J. Geophys. Research **66,** No. 3, 992–993 (Mar. 1961).

Microwave spectrum of isobutylene. Dipole moment, internal barrier, equilibrium conformation, and structure, V. W. Laurie, J. Chem. Phys. **34,** No. 5, 1516–1519 (May 1961).

Nonequilibrium processes in isotopically disordered crystals. Dependence on degree of order, R. Zwanzig, J. Math. Phys. **2,** No. 3, 370–372 (May–June 1961).

Intermolecular forces from optical spectra of impurities in molecular crystals, R. W. Zwanzig, Molecular Phys. **3,** No. 4, 305–311 (July 1960).

On the excitation of electromagnetic surface waves on a curved surface, J. R. Wait, IRE Trans. on Antennas and Propagation, **AP–8,** No. 4, 445–448 (July 1960).

Solubility product phenomena in hydroxyapatite-water systems, H. M. Rootare, V. R. Deitz, and F. G. Carpenter, Proc. 6th Tech. Session on Bone Char 1959, p. 287 (Bone Char Research Project Inc., Charlestown, Mass., 1961).

Reproducibilities of carbon and germanium thermometers at 4.2 °K, M. H. Edlow and H. H. Plumb, Proc. Cryogenic Eng. Conf., Boulder, Colo., Aug. 1960, **6,** 542–547 (1961).

The automatic chloride titrator in the analysis of some ionic constituents, M. D. Peiperl, F. G. Carpenter, and V. R. Deitz, Proc. 6th Tech. Session on Bone Char 1959, p. 53 (Bone Char Research Project Inc., Charlestown, Mass., 1961).

Spin-lattice relaxation in cerous magnesium nitrate, R. P. Hudson and R. S. Kaeser, Il Nuovo Cimento **19,** Serie X, 1275–1277 (Mar. 16, 1961).

The photolysis of ethyl vinyl ether, E. Murad, J. Am. Chem. Soc. **83,** 1327–1330 (1961).

Publications for which a price is indicated (except for Technical Notes) are available only from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. (foreign postage, one-fourth additional). Reprints from outside journals and the NBS Journal of Research may often be obtained directly from the authors.

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